EDIBLE POWDER MATERIAL HAVING EXCELLENT SHELF STABILITY

This invention relates to edible powder materials which have excellent shelf stability of a perfume, coloring agent and/or functional substance (e.g., vitamin) contained therein and which, when incorporated into various drinks, foods, cosmetics and the like, can impart a desired fragrance, flavor, color and/or function stably thereto for a long period of time. More particularly, it relates to powder compositions comprising at least one component selected from the group consisting of perfumes, coloring agents and functional substances, trehalose and water-soluble hemicellulose.

Conventionally, in order to impart a desired fragrance, flavor and/or color to drinks, foods, cosmetics and the like, and in order to impart thereto physiological activities such as a brain function-improving effect and a cholesterol-reducing effect, there have been commonly used powder materials obtained by mixing an oily material selected from oily perfumes, oil-soluble coloring agents and functional substances (e.g., vitamins), with a vegetable natural gum solution (e.g., a gum arabic solution) or with an emulsifier (e.g., modified starch or dextrin), an excipient and the like, and then spray-drying the resulting emulsified mixture. Alternatively, such powder materials may also be prepared by mixing or emulsifying an oily material as described above, with a synthetic surface-active agent (e.g., sucrose fatty acid ester, glycerol fatty acid ester or polyglycerol fatty acid ester), a suitable excipient and the like, and then spray-drying the resulting emulsified mixture.

However, when powder materials are prepared by emulsifying a perfume, coloring agent, and/or functional substance (e.g., vitamin) in the presence of an emulsifier, an excipient and the like, and then drying the resulting emulsified mixture, for example, by

spray drying, these powder materials are not always satisfactory from the viewpoint of the shelf stability of the fragrance, flavor, color and/or function.

Meanwhile, in order to improve the shelf stability of perfumes, coloring agents and functional substances (e.g., vitamins), the present inventors previously proposed a water-soluble powder perfume obtained by drying an emulsified mixture containing an edible oily material (e.g., perfume) and a water-soluble soybean polysaccharide (see Japanese Patent Laid-Open No. 107937/'95); a method for the preparation of a powdered perfume which comprises drying an emulsified mixture containing a perfume, trehalose, an emulsifier and water (see Japanese Patent Laid-Open No. 107911/'97); and a method for the preparation of a powdered functional substance which comprises drying an emulsified mixture containing a functional substance, trehalose, an emulsifier and water (see Japanese Patent Laid-Open No. 187249/'97).

The above-described propositions are considerably effective in improving the shelf stability of perfumes, coloring agents and functional substances (e.g., vitamins), but are not entirely satisfactory as yet.

The primary object of the present invention is to provide an edible powder composition which has excellent shelf stability of a perfume, coloring agent and/or functional substance (e.g., vitamin) contained therein and which can be used in various drinks, foods, cosmetics and the like to impart a desired fragrance, flavor, color and/or function stably thereto for a long period of time, without exerting any adverse influence on the inherent fragrance, flavor, color and taste thereof.

The present inventors made an intensive investigation in order to overcome the disadvantages of conventional edible powder materials as described above. As a result, it has now been found

that, by emulsifying a perfume, coloring agent and/or functional substance (e.g., vitamin) while using trehalose (i.e., a nonreducing disaccharide made up of two D-glucose molecules in α,α-1,1 linkage) obtained, for example, by the enzymatic degradation of starch, in combination with water-soluble hemicellulose, there can be obtained a powder composition having excellent shelf stability of the perfume, coloring agent and/or functional substance. Moreover, it has also been found that this powder composition can impart a desired fragrance, flavor, color and/or function stably to various drinks, foods, cosmetics and the like for a long period of time, without exerting any adverse influence on the inherent fragrance, flavor, color and taste thereof. The present invention has been completed on the basis of these findings.

Thus, the present invention provides a powder composition comprising at least one component selected from the group consisting of perfumes, coloring agents and functional substances, trehalose and water-soluble hemicellulose.

The present invention also provides a method for preparing the above-described powder composition which comprises drying an aqueous emulsion containing at least one component selected from the group consisting of perfumes, coloring agents and functional substances, trehalose and water-soluble hemicellulose.

The present invention will be more specifically described hereinbelow.

No particular limitation is placed on the types of the perfumes and coloring agents which can be used as raw materials in the present invention, and there may be used any of the perfumes and coloring agents which are commonly used in drinks, foods, cosmetics and the like. Usable perfumes include, for example, essential oils derived from citrus fruits such as orange, lemon, lime and grapefruit; vegetable essential oils such as flower essential oils,

peppermint oil, spearmint oil and spiced oil; powders, extracts, oleoresins, essences and recovery perfumes derived from cola nuts, coffee, vanilla, cocoa, black tea, green tea, oolong tea and spices; and synthetic perfume compounds, prepared perfume compositions and any mixtures thereof. Usable coloring agents include, for example, α-carotene, β-carotene, lycopene, paprika pigment, annatto pigment, chlorophyll, gardenia pigment, safflower pigment, monascus pigment, beet pigment, elderberry pigment, marigold pigment and cochineal pigment.

The term "functional substance" as used herein means any substance having a biological regulatory effect or physiological activity for mammals and, in particular, humans. Such functional substances include, for example, docosahexaenoic acid (DHA), eicosapentaenoic acid (EPA), DHA- and/or EPA-containing fish oil, linolic acid, γ-linolenic acid, α-linolenic acid, evening primrose oil, borage oil, lecithin, octacosanol, rosemary, sage, γ-oryzanol, β-carotene, palm carotene, perilla oil, chitin, chitosan, royal jelly and propolis; oil-soluble vitamins and their derivatives such as vitamin A, vitamin D, vitamin E, vitamin F and vitamin K; and water-soluble vitamins and their derivatives such as vitamin B_1, vitamin B_2, vitamin B_6, vitamin B_{12}, vitamin C, vitamin L, vitamin P, nicotinic acid, pantothenic acid and choline.

The trehalose used in the present invention may be prepared, for example, by culturing yeast in a glucose solution to produce trehalose in yeast cells, and then isolating the trehalose from the yeast cells; or by culturing a bacterium in a glucose solution to produce trehalose in the culture medium, and then separating and recovering the trehalose from the culture medium. However, any commercially available trehalose may also be used. The content of trehalose in the powder composition of the present invention is not strictly limited, but may be suitably chosen according to the type and

form of the perfume, coloring agent or functional substance used. However, trehalose is generally used in an amount of about 5 to about 90% by weight, preferably about 25 to about 85% by weight, and more preferably about 40 to about 80% by weight, based on the total weight of the powder composition.

The water-soluble hemicellulose used in the present invention is hemicellulose which has been made soluble in water by degrading it, for example, by subjecting it to proteolysis with a proteolytic enzyme or by heating it in an aqueous medium under acid conditions. The water-soluble hemicellulose may be derived from cereals and beans, such as soybeans, bean-curd refuse, corn and rice bran. Specific examples thereof include a powder product prepared by providing bean-curd refuse which is obtained as a by-product when bean curd is made from soybeans or when soybean protein is extracted from defatted soybean, homogenizing it with a homogenizer or the like, subjecting it to proteolysis with a protease or hydrolysis in the presence of an acid (for example, hydrolysis at a pH of about 3 to about 7, preferably about 4 to about 5, and a temperature of about 100 to about 150°C, preferably about 110 to about 120°C), separating a water-soluble component by centrifugation or filtrating, and drying it by drying means such as spray drying; and a powder product prepared in the same manner as described above, except that a low-molecular fraction is removed from the water-soluble component. More specifically, there may be used water-soluble hemicellulose derived from a soybean cotyledon, which can be produced by subjecting a soybean cotyledon to heat extraction in an acidic region as described in U.S. Patent 5,700,397 (= EP 0 598 920 B1). Alternatively, commercial products of water-soluble hemicellulose may also be used, and an example thereof is "SOYAFIVE-S" that is sold by Fuji Oil Co., Ltd., Osaka, Japan.

The water-soluble hemicellulose, which may be prepared

by any of the above-described methods, contains rhamnose, fucose, arabinose, xylose, galactose, glucose and uronic acid as constituent sugar components, and may further contain mannose and fructose in some cases. Although the contents of these constituent sugar components may vary according to the type of the raw material and the method of preparation, it is usually preferable that the water-soluble hemicellulose used in the present invention contain 1 to 5% by weight of rhamnose, 2 to 8% by weight of fucose, 15 to 50% by weight of arabinose, 4 to 10% by weight of xylose, 25 to 50% by weight of galactose, not greater than 4% by weight of glucose, and 15 to 25% by weight of uronic acid. Moreover, the water-soluble hemicellulose may generally have an average molecular weight of about 50,000 to about 1,000,000, preferably about 100,000 to about 700,000, as measured in a 0.1M $NaNO_3$ solution by the limiting viscosity method using standard pullulan.

The content of water-soluble hemicellulose in the powder composition of the present invention is not strictly limited, but may vary according to the type of the perfume, coloring agent or functional substance used, the use of the powder composition of the present invention, and the like. However, water-soluble hemicellulose is generally used in an amount of about 1 to about 80% by weight, preferably about 5 to about 60% by weight, and more preferably about 10 to about 40% by weight, based on the total weight of the powder composition.

No particular limitation is placed on the ratio in which trehalose and water-soluble hemicellulose are present in the powder composition of the present invention. However, a powder composition having excellent shelf stability of the perfume, coloring agent and/or functional substance (i.e., vitamin) contained therein is obtained when the weight ratio of trehalose to water-soluble hemicellulose is in the range of about 50 : 1 to about 1 : 50, preferably

about 30 : 1 to about 1 : 25, and more preferably about 8 : 1 to about 1 : 1.

The powder composition of the present invention can readily be prepared by mixing at least one component selected from among the above-described perfumes, coloring agents and functional substances, with trehalose, water-soluble hemicellulose and water, and drying the resulting mixture. If necessary, the aforesaid composition may further contain sugars such as sucrose, lactose, glucose, starch syrup and reduced starch syrup; sugar alcohols; various starch hydrolyzates and starch derivatives (e.g., dextrin); starch; gelatin; natural gums such as gum arabic; and the like. The contents of these additives may be suitably chosen according to the properties desired for the powder composition, and the like.

One preferred embodiment of the method for preparing the powder composition of the present invention is as follows. First of all, trehalose and water-soluble hemicellulose as described above are dissolved in water. Then, at least one component selected from among the above-described perfumes, coloring agents and functional substances is added thereto and mixed therewith by means of a homomixer, colloid mill, high-pressure homogenizer or the like. The resulting emulsion is dried by drying means such as vacuum drying, spray drying or freeze drying. Thus, there can be obtained a powder composition having excellent shelf stability of the perfume, coloring agent or functional substance.

The powder compositions obtained in the above-described manner may be incorporated, in appropriate amounts, into drinks, powdered drinks and foods such as chewing gum, tablet candies, snacks, processed marine products, processed meat products, retort foods, frozen foods, instant noodles and health foods, thus providing drinks and foods to which a desired fragrance, flavor, color and/or function is imparted stably for a long period of time. Moreover, they

may be incorporated, in appropriate amounts, into cosmetics such as antiperspirants, shampoos, hair creams, pomades, face powder and lipsticks, thus providing cosmetics to which a desired fragrance, color and/or function is imparted stably for a long period of time. Furthermore, they may also be used in sanitary and hygienic materials such as washing detergents, disinfectants and room aromatics; pharmaceutical preparations; tobacco; and the like.

When the powder compositions are incorporated into drinks, foods, cosmetics and the like, their amount used may vary according to the type and form of the product being processed. However, the powder compositions are generally used in an amount of about 0.001 to about 0.1 part by weight, preferably about 0.01 to about 0.05 part by weight, per 1 part by weight of the product being processed.

The present invention is more specifically explained with reference to the following examples, comparative examples and reference examples.

Example 1

20 g of water-soluble hemicellulose (SOYAFIVE-S LA200, manufactured by Fuji Oil Co., Ltd.; with an average molecular weight of about 200,000) and 60 g of trehalose were added to and dissolved in 100 g of water. This solution was sterilized by heating at 85-90°C for 15 hours. After it was cooled to 40°C, 20 g of a lemon flavor was added thereto and mixed therewith. The resulting mixture was emulsified with a TK-Homomixer (trade name; manufactured by Tokushu Kika Kogyo Co., Ltd.). Using a Mobile Minor type spray dryer (manufactured by Niro Inc.), this emulsion was spray-dried at an inlet temperature of 150°C and an outlet temperature of 80°C to obtain 95 g of a lemon powder perfume (inventive product 1).

Example 2

The procedure of Example 1 was repeated, except that

the amount of trehalose was altered from 60 g to 40 g, and 20 g of gelatin hydrolyzate was additionally used. Thus, there was obtained 95 g of a lemon powder perfume (inventive product 2).

Comparative Example 1

The procedure of Example 1 was repeated, except that the amount of water was altered from 100 g to 150 g, and 80 g of gum arabic was used in place of 20 g of water-soluble hemicellulose and 60 g of trehalose. Thus, there was obtained 90 g of a lemon powder perfume (comparative product 1).

Comparative Example 2

The procedure of Example 1 was repeated, except that the amount of water was altered from 100 g to 120 g, and 40 g of gum arabic and 40 g of dextrin (DE10) were used in place of 20 g of water-soluble hemicellulose and 60 g of trehalose. Thus, there was obtained 95 g of a lemon powder perfume (comparative product 2).

Reference Example 1

According to the procedure described below, tablets were prepared by adding 0.5% of each of the lemon powder perfumes obtained in Examples 1 and 2 and Comparative Examples 1 and 2. The tablets so prepared were subjected to a storage test as described below, and then organoleptically examined for fragrance and flavor by expert panelists. The results thus obtained are shown in Table 1.

Method for the preparation of tablets

(Formulation)

Raw materials	Amount used
1. Powder sugar	903 g
2. Lactose	30
3. Vitamin C	37
4. Citric acid powder	15
5. 1% aqueous solution of gelatin	40

6. Sucrose fatty acid ester	10
7. Lemon powder perfume	5
Total	1,040
Dry weight	1,000

(Procedure)

(1) After powder materials 1 to 4 are mixed, material 5 is added thereto and this mixture is agitated until it becomes homogeneous.

(2) The resulting blend is granulated to a size of less than 30 mesh.

(3) The granules are dried at 45°C for 60 minutes.

(4) Materials 6 and 7 are added to and mixed with the granules dried in (3).

(5) The resulting blend is tableted under the following conditions.

Weight: 1.8 g/tablet
Diameter: 2 cm
Pressure: 40 kg/cm^2/tablet

Storage test method

(1) Tablets were put into a low-density polyethylene bag, which was stored in the dark at 50°C for 4 weeks.

(2) Tablets were put into a high-density polyethylene bag, which was stored under fluorescent lamp illumination at 4,500 lux for 2 weeks.

As a control, tablets were put into an aluminum bag, which was stored at -18°C.

Table 1

Results of Organoleptic Examination

	After storage at -18°C (control)	After storage in the dark at 50°C for 4 weeks	After storage at 4,500 lux for 2 weeks
Inventive product 1	10	9	8
Inventive product 2	10	8	7
Comparative product 1	10	3	2
Comparative product 2	10	2	2

The numerals given in the above table are relative values obtained by comparing the tested tablets with the control tablets stored at -18°C which are rated as 10. Smaller values indicate that the fragrance and flavor were lose to a higher degree.

As is evident from Table 1, the tablets of Examples 1 and 2 having a powder composition of the present invention incorporated thereinto are superior in the stability of fragrance and flavor to the tablets of Comparative Examples 1 and 2.

Scanning election micrographs (magnification: ×2000)

Scanning election micrographs (×2000) of particles of the lemon powder perfumes prepared in Example 1 and Comparative Example 1 are given in FIGs. 1 and 2, respectively. A comparison of FIG. 1 with FIG. 2 reveals that the particle of the lemon powder perfumes of Example 1 in accordance with the present invention is in the form of a dense spherule having a stiff wall surface, and hence exhibits excellent stability of the lemon perfume contained therein.

Example 3

20 g of water-soluble hemicellulose (SOYAFIVE-S

LA200, manufactured by Fuji Oil Co., Ltd.; with an average molecular weight of about 200,000) and 60 g of trehalose were added to and dissolved in 100 g of water. This solution was sterilized by heating at 85-90°C for 15 hours. After it was cooled to 60°C, 20 g of l-menthol was added thereto and mixed therewith. The resulting mixture was emulsified with a TK-Homomixer. Using a Mobile Minor type spray dryer (manufactured by Niro Inc.), this emulsion was spray-dried at an inlet temperature of 160°C and an outlet temperature of 80°C to obtain 95 g of a menthol powder perfume (inventive product 3).

Example 4

The procedure of Example 3 was repeated, except that the amount of trehalose was altered from 60 g to 40 g, and 20 g of gelatin hydrolyzate was additionally used. Thus, there was obtained 95 g of a menthol powder perfume (inventive product 4).

Comparative Example 3

The procedure of Example 3 was repeated, except that the amount of water was altered from 100 g to 150 g, and 80 g of gum arabic was used in place of 20 g of water-soluble hemicellulose and 60 g of trehalose. Thus, there was obtained 90 g of a menthol powder perfume (comparative product 3).

Comparative Example 4

The procedure of Example 3 was repeated, except that the amount of water was altered from 100 g to 120 g, and 40 g of gum arabic and 40 g of dextrin (DE10) were used in place of 20 g of water-soluble hemicellulose and 60 g of trehalose. Thus, there was obtained 90 g of a menthol powder perfume (comparative product 4).

Reference Example 2

According to the same procedure as described in Reference Example 1, tablets were prepared by adding 0.5% of each of the menthol powder perfumes obtained in Examples 3 and 4 and Comparative Examples 3 and 4. The tablets so prepared were subjected

to a storage test as described below, and then visually examined for the separating-out of menthol. The results thus obtained are shown in Table 2.

Storage test method

Tablets were put into a low-density polyethylene bag, which was stored in the dark at 50°C for 2, 4 or 12 weeks.

Table 2

Separating-out of Menthol

	50°C, 2 weeks	50°C, 4 weeks	50°C, 12 weeks
Example 3	No	No	No
Example 4	No	No	No
Comparative Example 3	No	Yes	Yes
Comparative Example 4	No	Yes	Yes

As is evident from Table 2, the tablets of Examples 3 and 4 having a powder composition of the present invention incorporated thereinto are superior in the stability of menthol to the tablets of Comparative Examples 3 and 4.

Example 5

20 g of water-soluble hemicellulose (SOYAFIVE-S LA200, manufactured by Fuji Oil Co., Ltd.; with an average molecular weight of about 200,000) and 70 g of trehalose were added to and dissolved in 100 g of water. This solution was sterilized by heating at 85-90°C for 15 hours. After it was cooled to 40°C, 10 g of paprika oil was added thereto and mixed therewith. The resulting mixture was emulsified with a TK-Homomixer. Using a Mobile Minor type spray

dryer (manufactured by Niro Inc.), this emulsion was spray-dried at an inlet temperature of 160°C and an outlet temperature of 80°C to obtain 95 g of a paprika-containing powder (inventive product 5).

Comparative Example 5

The procedure of Example 5 was repeated, except that the amount of water was altered from 100 g to 150 g, and 30 g of gum arabic and 60 g of dextrin (DE10) were used in place of 20 g of water-soluble hemicellulose and 70 g of trehalose. Thus, there was obtained 90 g of a paprika-containing powder (comparative product 5).

Reference Example 3

The paprika-containing powders obtained in Example 5 and Comparative Example 5 were subjected to a storage test as described below. Thereafter, their contents of paprika pigment were measured with a spectrophotometer. The results thus obtained are shown in Table 3.

In Table 3, the retention of paprika pigment after storage at 50°C for 4 weeks is expressed as a percentage based on the content of paprika pigment (= 100%) immediately after preparation.

Storage test method

Each of the paprika-containing powders (i.e., inventive product 5 and comparative product 5) was put into a low-density polyethylene bag, which was stored in the dark at 50°C for 4 weeks.

Table 3

Retention of Paprika Pigment

	After storage at 50°C for 4 weeks
Example 5	97.5%
Comparative Example 5	32.8%

Example 6

20 g of water-soluble hemicellulose (SOYAFIVE-S LA200, manufactured by Fuji Oil Co., Ltd.; with an average molecular weight of about 200,000) and 76.5 g of trehalose were added to and dissolved in 100 g of water. This solution was sterilized by heating at 85-90°C for 15 hours. After it was cooled to 40°C, 3 g of vitamin A palmitate and 0.5 g of vitamin E were added thereto and mixed therewith. The resulting mixture was emulsified with a TK-Homomixer. Using a Mobile Minor type spray dryer (manufactured by Niro Inc.), this emulsion was spray-dried at an inlet temperature of 160°C and an outlet temperature of 80°C to obtain 95 g of a vitamin A-containing powder (inventive product 6).

Comparative Example 6

The procedure of Example 6 was repeated, except that the amount of water was altered from 100 g to 150 g, and 40 g of gum arabic and 56.5 g of dextrin (DE10) were used in place of 20 g of water-soluble hemicellulose and 76.5 g of trehalose. Thus, there was obtained 90 g of a vitamin A-containing powder (comparative product 6).

Reference Example 4

The vitamin A-containing powders obtained in Example 6 and Comparative Example 6 were subjected to a storage test as described below. Thereafter, their contents of vitamin A were measured by high-performance liquid chromatography. The results thus obtained are shown in Table 4.

In Table 4, the retention of vitamin A after storage at 35°C for 4 weeks is expressed as a percentage based on the content of vitamin A (= 100%) immediately after preparation.

Storage test method

Each of the vitamin A-containing powders (i.e., inventive product 6 and comparative product 6) was put into a low-density

polyethylene bag, which was stored in the dark at 35°C for 4 weeks.

Table 4

Retention of Vitamin A

	After storage at 35°C for 4 weeks
Example 6	94.5%
Comparative Example 6	12.0%

As described above, the powder compositions of the present invention have excellent shelf stability of the perfume, coloring agent and/or functional substance contained therein, and can be used in various drinks, foods, cosmetics and the like to impart a desired fragrance, flavor, color and/or function stably thereto for a long period of time, without exerting any adverse influence on the inherent fragrance, flavor, color and taste thereof. Accordingly, the powder compositions of the present invention are very useful in that they can be expected to have a wide range of applications including drinks, foods, cosmetics and the like.